

# Water Determination in Biodiesel

## VOLUMETRIC KARL FISCHER TITRATION

### Introduction

For the determination of water content in biodiesel samples according to Volumetric Karl Fischer.

### Apparatus

- TitroLine® 7500 KF or TitroLine® 7750/7800 with KF accessories
- µl syringe with needle

### Electrode

- **Electrode:** KF1100

### Reagents

- **Solvent:** Methanol/chloroform mixture 1:2 (one component), solvent oil, solvent CM or solvent crude oil, or a mixture of 10 ml solvent and 20 ml chloroform
- **Standardization:** With di-sodiumtetratrate dihydrate p.a., pure water or liquid standard
- **Titrant:** Single or two-component reagent (1 ml = ca. 2 mg)



## Procedure

Select the method: **Oil 1 component**

The titration parameters can be adapted to the sample. In many cases, the default parameters can be used without any change, but some samples need a longer extraction time (default = 30 seconds) or a longer minimum titration time.

The solvent is added with the pump. Subsequently, the KF method is started with F1/Start and the solvent is automatically conditioned. Press **<START>** when conditioning is complete and insert the sample ID. Press **<ENTER>**.

Fill a 2 - 10 ml syringe with the biodiesel sample, keeping some air in the tip of the needle to avoid loss of sample. Use a needle with a diameter > 1 mm if possible.

Place the syringe on an analytical balance. Use a beaker and stand the needle upwards in the beaker. Press the tare button on the balance. Introduce the sample through the septum into the vessel.

Place the syringe on the balance again and record the absolute weight from the balance display. Press the print button of the balance if the balance is connected to the titrator, or use the keyboard to insert the weight into the TitroLine. Confirm with **<ENTER>**.

The TitroLine automatically starts the titration, and results will be displayed on the screen upon completion.

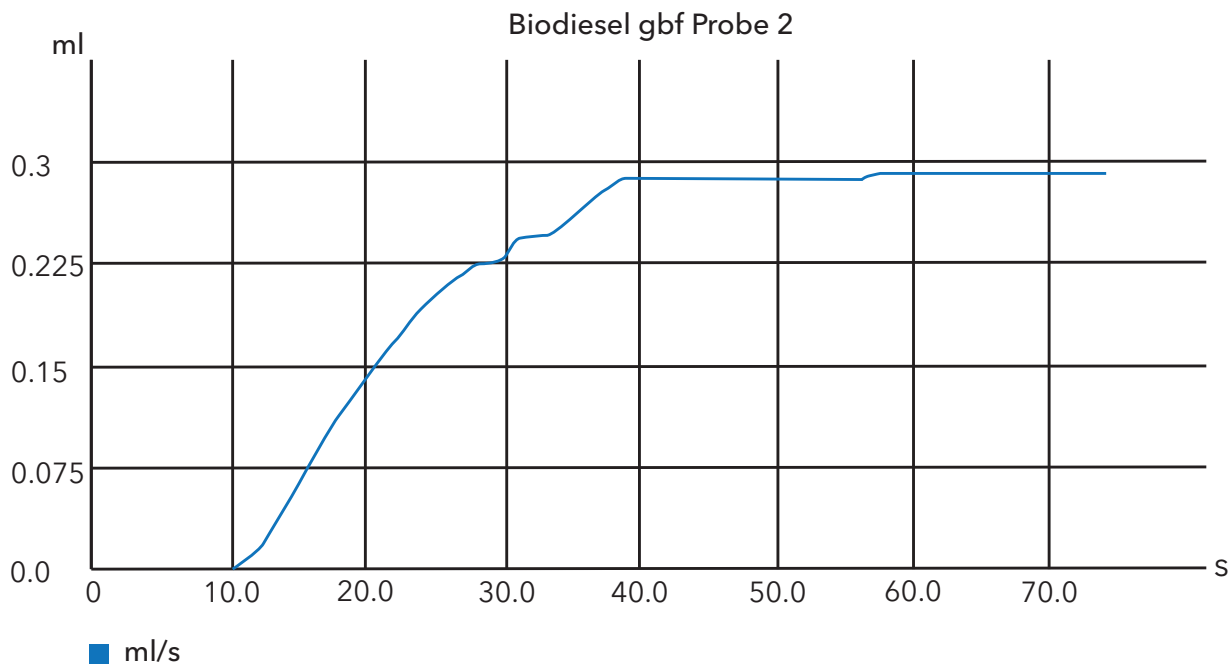
### Electrode Handling

You can store the double platinum electrodes wet (in the titration vessel) or dry.

Method Example (Page 1)

GLP documentation

Titration graph



Method data

Method name:	Sample 2-Comp.	Titration duration:	1 m 13 s
End date:	22.01.15	End time:	17:00:26

Titration data

Sample ID:	Biodiesel gbf Probe 2	Weight:	6.31580 g
Start $\mu$ A:	1.202 $\mu$ A	End $\mu$ A:	21.309 $\mu$ A
Start drift:	7 $\mu$ g/min	End drift:	41 $\mu$ g/min
Consumption:	0.292 ml	Drift correction:	Off
Water:	0.0217 %		

Calculation formula

Water:	$(EP-B)*T*M*F1/(W*F2)$	Mol (M):	1.00000
Blank value (B):	0.0000 ml	Titre (T):	4.6828 (a)
Factor 1 (F1):	0.1000	Weight (W):	6.31580 g (a)
Factor 2 (F2):	1.0000	Statistics:	Off

## Method Example (Page 2)

### Method data

Method name:	Oil 1-Comp.	Created at:	05/25/16 14:26:48
Method type:	Automatic titration	Last modification:	05/25/16 14:33:13
Titration mode:	KF	Documentation:	GLP
Linear steps:	0.005 ml		
Fixed delay time:	1 s		
Extraction time:	30 s		
Pretitration:	Off		
Endpoint:	20.0 $\mu$ A		
Delta endpoint:	5.0 $\mu$ A		
Endpoint delay:	10 s		
Drift:	100 $\mu$ g/min	Stirrer control:	free
Drift correction:	Off		
Min. titration time:	120 s		
Max. titration time:	600 s		
Polarization voltage:	100 mV		
<u>Dosing parameter</u>			
Dosing speed:	60.00 %	Filling speed:	30 s
Maximum dosing volume:	50.00 ml		
<u>Calculation formula</u>			
Water:	$(EP-B)*T*M*F1/(W*F2)$	Mol (M):	1.00000
Unit:	ppm	Decimal places:	1
Blank value (B):	0.0000 ml	Titre (T):	auto
Factor 1 (F1):	1000.0000	Weight (W):	man
Factor 2 (F2):	1.0000	Statistics:	Off

Hydranal®-Workshop - Water Reagents according to Eugen Scholz for Karl-Fischer titration, Riedel-de-Haën.



Xylem Inc.  
1725 Brannum Lane  
Yellow Springs, Ohio 45387

Application/Technical Support:  
+ 1-845-258-1200  
[titration.yesi@xylem-inc.com](mailto:titration.yesi@xylem-inc.com)

Ordering:  
+1-937-767-7241  
[orders@ysi.com](mailto:orders@ysi.com)